



## PATENT SPECIFICATION

NO DRAWINGS

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## COMPLETE SPECIFICATION

## Base Web for Casing and method of making same

We, THE DEXTER CORPORATION, formerly C. H. Dexter & Sons Incorporated, A Corporation organised and existing under the Laws of the State of Connecticut, United States of America, of Windsor Locks, Connecticut, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to reinforced film or casing material used for packaging meat products. More particularly it relates to an improved base web and a casing material made therefrom as well as to a new and improved method of producing such webs and materials.

Heretofore it has been the practice to make reinforced films, tubings, and casings for meat products by sequential and repeated treatment of fibrous papers or webs with a cellulose coating material, such as a "Viscose" (Registered Trade Mark) solution. That treatment includes the preparation of a base material or web having substantial caustic stability followed by the final casing-forming operations. The preparation of the base web is effected by treating a preformed and dried paper with a dilute "Viscose" solution followed by the steps of drying, regenerating the cellulose, washing with fresh water and redrying while the casing-forming operations comprise the steps of impregnating the base web with a highly caustic "Viscose" solution, regeneration of the impregnate, washing and drying of the final casing. In short, at least four forming operations followed by four drying steps were required. The initial application of the dilute "Viscose" solution is required for imparting to the base paper web sufficient caustic resistance to retain its structural integrity during the final casing-forming operations wherein the highly alkaline conditions are employed. It is also necessary

that the initially treated web retain its porous, absorbent characteristics in order to permit impregnation during the final "Viscose" coating operation. This process, resulting in a fiber reinforced, leak-proof, cellulosic film material, is set forth in greater detail in U.S. Patent No. 3,135,613 recently issued to William F. Underwood and entitled "Impregnated Paper Webs and Methods of Making Sausage Casing Thereof".

In view of the need for a casing material which will withstand excessive stuffing pressures without bursting yet is subject to a known, substantially uniform variation during automatic packaging operations regardless of the portion of base web employed, great emphasis has been placed on the need for improving and controlling the burst strength of the casings as well as the transverse elongation profile of the base web. Such control is particularly important where the products are automatically made on modern equipment necessitating greater strength and reliability in the casing. Additionally the automatically sliced and wrapped product must meet a stated meat content and any increase in the diameter of the casing over the minimum required necessarily results in a loss to the meat packer. Although base webs made according to the known method have yielded casings of satisfactory burst strength for the equipment utilized heretofore, modern machinery has required casings of improved burst strength.

The burst strength of the impregnated casing material renders it particularly well suited for packing meat products under pressure since it can provide firm, uniform enclosures, tubings or casings for meat products such as sausage or bologna. These casings are generally made by cutting the long base paper webs into longitudinal strips and then forming them into long cylinders by curving the strips about their longitudinal axes and providing an overlapping seam. It will be appreciated that some cylinders will be formed

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from the edge portions of the base web while others will be formed from the center or intermediate portions thereof. Consequently, unless the base webs possess a substantially uniform or non-varying transverse elongation profile across the width thereof, the tubes or cylinders made from the center portions will vary from those made from the edge portions. As a result it frequently occurred that the desired casing material was produced from only the central portions of the base web because of such variation. The casings were produced by impregnating the web with a highly caustic "Viscose" solution, passing the impregnated film or cylinder through regenerating baths, and finally washing and drying the casing.

Accordingly, it is an object of the present invention to provide a base web having an improved transverse elongation profile and a reinforced film or casing made therefrom possessing improved burst strength, such film being suitable for use in the packaging of meat products.

Another object of the present invention is to provide a base web material made on wide papermaking machines, which web is free from appreciable variation in its transverse elongation profile over the entire width of the web and additionally exhibits satisfactory strength and resistance to strong caustic solutions, thereby rendering it suitable for use in casing formation.

A further object of the invention is to provide a reinforced film or casing adapted for the packaging of meat products and having both improved burst strength and more effective bonding between the fibers of the base web and the cellulosic coating.

A still further object is to provide a casing of improved burst strength made from base webs having an appreciably lower tensile strength than webs utilized for this purpose heretofore.

Still another object of the present invention is to provide an improved process for producing the base web and casing as set forth above, which process eliminates some of the variation-producing operations utilized heretofore in the preparation of web and casing materials and results in a more uniform base web and a casing of improved burst strength.

The term "base web" as used herein refers to fibrous, caustic stable, paperlike materials prior to being treated with the highly alkaline "Viscose" solution, such materials being preferably formed on papermaking machines.

According to the present invention there is provided a method of producing reinforced films suitable for packing meat products comprising the steps of forming a dilute dispersion of cellulosic fibers and depositing said dispersed fibers in the form of a continuous

web while incorporating an alkaline-curing synthetic resin impregnating the web with a caustic cellulose solution; the web having a density prior to impregnation of at least 0.15 g/cc as determined by TAPPI test method T411—m—44; regenerating the cellulose impregnating said web; and thereafter drying the impregnated web to provide a reinforced film-like material.

The invention also consists in a reinforced film suitable for packing meat products comprising a fibrous base web impregnated with regenerated cellulose, said base web comprising essentially cellulosic, randomly interconnected fibers and having an alkaline-curing synthetic resin incorporated therein and a density prior to impregnation of at least 0.15 g/cc, as determined by TAPPI test method T411—m—44.

Briefly the process comprises the steps of forming a dilute suspension of cellulosic fibers and thereafter forming a base web from the suspension while incorporating into the web certain alkaline-curing resin material, such as polyethylene imine and preferably the polymeric reaction products of epichlorohydrin and a polyamide. The incorporation of the resin may be effected by addition thereof at the beater or the head box. The resultant fiber base web or paper possesses a desirable tensile ratio, a uniform distribution of fibers across the entire width of the web and, although exhibiting a lower tensile strength, results in a casing of improved burst strength and more effective bonding between the fibers of the web and the viscose coating. It is also an advantage of the present invention that the incorporation of the resin into furnish will not hamper the uniform deposition of the fibers on the screen or wire of the papermaking machine, particularly when employing the inclined screen arrangement more fully described in U.S. Patent No. 2,045,095, issued June 23, 1936 to Fay H. Osborne. The web thus formed is preferably wet pressed and exhibits the required caustic resistance and structural stability to undergo casing formation. Further, the web exhibits substantially less variation in its transverse direction elongation profile and therefore less variation between cylindrical casings made from the center and the edges of the base web. The web is then coated, preferably on both sides, with a regeneratable cellulose derivative such as the cellulose ether solution known as "Viscose" and the coating is permitted to thoroughly impregnate the web. Subsequently the impregnant is regenerated, and the reinforced film resulting therefrom is washed and dried.

The base web for the reinforced film or casing is generally composed of natural fibers of pure cellulose and preferably comprises the long, lightweight and nonhydrated fibers of the *Muss texilis* species, typical of which

are hemp fibers. Webs made from this material are generally soft porous papers of uniform texture and thickness and possess a tensile ratio close to unity; that is, the tensile strengths in the machine and transverse directions are substantially the same. However, it will be appreciated that tensile ratio may vary from about 0.5 to about 1.0 where such is desired.

- 10 The fibers in their naturally occurring condition are held or bonded together by non-cellulosic material which includes lignin, gums, and waxes. It is therefore necessary to free these fibers, which are more or less uniform in length, generally cylindrical in shape, and substantially free from lint, in a non-hydrating manner from the non-cellulosic binders found in the raw stock. This may be accomplished by chemically digesting the stock and facilitating separation by stirring without mechanical pressure, thereby preventing hydration or change in the physical characteristics of the fibers. The digestion may be accomplished according to known techniques
- 25 utilizing strong alkaline solutions, such as solutions containing, *inter alia*, caustic soda, for a long duration of time under high pressures and at elevated temperatures. The digested fibers may then be washed free of the caustic and dispersed so as to form the dilute fiber suspension used to form the base web.

- It is desirable that the alkaline-curing resin which is incorporated in the base web according to the present invention not only imparts to the web a resistance to caustic but also provides little or no interfering with the absorption characteristics of the paper. Preferably the resin should improve the bonding between the fibers of the web and the "Viscose" coating since it is believed that the latter quality results in a substantial improvement in the burst strength of the casing. At the same time, it should be borne in mind that the paper web should be devoid, at least as far as possible, of impregnants which might interfere with both the absorption and bonding mechanisms. The resin utilized should effectuate as little resistance as possible to the penetration of the viscose coating into the web.

- Alkaline-curing resins such as polyethylene imine have proved satisfactory from the standpoint of caustic resistance and transverse elongation profile characteristics and have resulted in burst ratios for the casings which are comparable to those obtained by the previously employed dilute "Viscose" treatment. However, the preferred resinous materials are the uncured thermosetting resins, and particularly, the polymeric reaction products of epichlorohydrin and polyamides containing secondary amine groups. Preferably the epichlorohydrin acts as a cross-linking agent and should be used in sufficient amounts to convert all the

secondary amine groups to tertiary amine groups, i.e. from 1.0—1.5 moles for each secondary amine group of the polyamide. Generally polyamides derived from polyalkylene polyamines and saturated or unsaturated aliphatic or aromatic polycarboxylic acids containing from 3 to 10 carbon atoms are preferred. A typical example of such material is the water-soluble epichlorohydrin-polyamide reaction product sold by Hercules Powder Company, Wilmington, Delaware, under the name "Kymene 557" (Registered Trade Mark).

Although the resin additive may be incorporated at any suitable step in the manufacturing process of the base web, it has been found highly effective when the material is added to the beater thereby permitting sufficient intermixing and dispersing thereof prior to the web formation. Post web formation incorporation of the resin may also be employed with success. The amount of resin used will vary somewhat depending upon the desired properties, as well as the point of addition; however, good results have been obtained when utilizing up to 10.0% by weight, and preferably 0.1% to 5.0% of the solids in the furnish. For example, when used as a beater additive, an amount of 0.5% to 3.0% by weight based upon the solids within the furnish may be employed while post web formation treatments generally use a higher range of 1.0% up to 5.0% by weight or higher.

The base web as formed from the dilute dispersion and containing the resin in accordance with the present invention may be then wet-pressed by passing the web through a pair of pressure rolls prior to the complete drying thereof. The thus formed and pressed web has a strength high enough so that it can maintain its structural integrity during subsequent operations yet not being so dense as to prevent the penetration of the highly caustic viscose solution into the interstices of the web. Generally, such webs possess a density of 0.15 g/cc or more according to TAPPI test method T411—m—44 although webs exhibiting a density above 0.35 g/cc are undesirable due to their low absorption of the "Viscose" solution.

The transverse elongation profile of the base web is determined by taking samples of the web from numerous positions across the extent thereof. As will be appreciated the largest variation is observed between the edge and center portions thereof. The samples used for testing are rectangular strips with their longitudinal direction being along the cross direction of the web. The dry samples are placed in an "Instron" (Registered Trade Mark) tensile tester made by Instron Engineering Corporation and the length of the sample between the jaws is accurately measured. Usually the strips being tested are 1" wide

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and provide a longitudinal test distance of about 2½". The dry samples are then stretched longitudinally at a constant straining rate of about 20% per minute while recording the force required as a function of sample length. The percentage of elongation is calculated for each sample.

The pressed base web after being dried in a conventional manner is ready for the casing-forming operations including the impregnation with the highly caustic "Viscose" solution. As discussed in the Underwood patent mentioned hereinbefore, the web is treated internally and externally with the "Viscose" solution either before, during or after tube formation. The solution employed may be the standard alkaline solution of regeneratable cellulose derivatives comprising about 6% by weight of sodium hydroxide and 7% by weight of cellulose. The impregnated web, strip or cylindrical tube is then passed through an aqueous regenerating bath prior to its final wash with fresh water and subsequent drying. The regeneration bath is generally a dilute mineral acid solution, for example, a solution containing 1% to 4% by weight of sulfuric acid and 15% to 25% sulfate salts.

The casing cylinders made in this manner generally exhibit an improvement in burst properties of about 10% or more over casings made in accordance with the process described in the Underwood patent. It is additionally significant that this improvement is effected according to the present invention when using base webs of substantially lower tensile strength. It will, of course, be appreciated that absolute values for the burst pressure will vary depending on both the ream weight of the web material and the diameter of the cylindrical casing, higher values being obtained from the smaller diameter cylinders. The burst pressures are obtained by inflating water soaked cylinders with air until they rupture or burst and measuring the pressure attained during the operation. For comparative purposes the variables are effectively removed by always testing cylinders of uniform size and relying on burst ratios rather than pressures, the ratio being the pressure at the time of burst divided by the ream weight for the base web material being tested.

The term "ream weight" as used herein refers to the weight in pounds of 480 sheets, each being 24" wide and 36" long in accordance with the TAPPI test method T410—os—61 or its related standard.

Having generally described the invention, the following examples some of which are comparative are included for purposes of illustration so that the invention may be more readily understood and are in no way intended to limit the scope of the invention unless otherwise specifically indicated. All amounts are on a weight basis unless otherwise specified.

#### EXAMPLE 1 (Comparative)

A furnish consisting of a dilute dispersion of 100% hemp fibers was formed into a continuous web and dried. The hemp paper thus formed was treated with a 1% solution of "Viscose" made by diluting in an appropriate manner a "Viscose" solution containing 6% by weight NaOH and 7% by weight cellulose. Following the procedure outlined in the Underwood patent mentioned hereinbefore, a cellulose material comprising about 96.6% alpha cellulose was employed. The treated paper was dried to a moisture content below 5% after which it was passed through a regenerating sulfuric acid bath, washed with fresh water and dried. Samples taken from the center and the edges of the resultant web were tested for dry transverse elongation using a 4 pound load. The tensile strengths of the base web were also determined and the results of these tests are given in Table I.

Casings were then made from this web in the conventional manner by impregnation with a highly caustic "Viscose" solution followed by regeneration and drying. The "Viscose" solution employed contained 6% by weight sodium hydroxide and 7% by weight cellulose (about 98% alpha cellulose content). The prepared casings were then subjected to burst pressure testing by inflating a water soaked casing with air until it burst. The results of this test are also set forth in Table I wherein the burst ratio is the pressure employed divided by the ream weight of the base web.

#### EXAMPLE 2.

Utilizing a portion of the 100% hemp fiber furnish of Example I there was dispersed therein at the beater about 1.25% by weight based on the total solids content of the furnish of polyethylene imine resin ("Chemical WSA—300" from Chemirad Corporation). A web was formed using substantially the same machine conditions as in Example I. The web thus formed was wet pressed and dried, after which samples were taken from the center and edges thereof for transverse elongation tests. Tensile strength determinations were also made.

Casings of the same diameter as Example I were then made from this web in the same manner as set forth in Example I and tested for burst strength. The results of these tests are set forth in Table I.

#### EXAMPLE 3.

Example 2 was repeated except that 1.00% by weight of a polymeric reaction product of epichlorohydrin and a polyamide ("Kymene 557") (Registered Trade Mark) was substituted for the polyethylene imine resin. The test results are given in Table I.

TABLE I

Test	Example 1	Example 2	Example 3
<u>Wet Breaking Strength</u>			
(g/in width)			
Machine Direction —	1048	577	829
Cross Direction —	1048	594	783
<u>Dry Breaking Strength</u>			
(g/in width)			
Machine Direction —	4492	3217	3283
Cross Direction —	3979	2454	2358
Density (g/cc)	0.251	0.314	0.322
<u>Transverse Elongation</u>			
Edge (% Elongation)	4.19	3.35	3.37
Center (% elongation)	1.39	1.50	1.89
Reduction	—	34%	47%
Burst Pressure (mm)	414	418	475
Ream Weight (lbs)	12.8	12.8	12.5
Burst Ratio	32.3	32.7	38.0
Improvement in Burst Strength	—	1.24%	17.65%

From a comparison of the transverse elongation results given in Table I it can be readily seen that the base webs made according to the present invention using both polyethylene imine and the epichlorohydrin reaction product exhibited a substantial reduction in the elongation differential between the center and edges of the web, the latter resin reducing the differential by almost 50%. Additionally, casings made from these webs exhibited burst ratios which were as high as (see Example 2) or higher than (see Example 3) the casings made according to the conventional technique despite a substantial decrease in the tensile or breaking strength of the base webs utilized.

#### EXAMPLE 4. (Comparative)

The procedure employed in Example 1 was repeated for Example 4 although a different furnish of hemp fibers was employed.

#### EXAMPLE 5.

Using a portion of the 100% hemp fiber furnish of Example 4 there was dispersed in the furnish at the beater about 1.00% by weight based on the total solids content of the furnish of a polymeric reaction product of epichlorohydrin and a polyamide ("Kymene 557"). A base web was formed utilizing substantially the same machine conditions as in Example 4, after which samples were taken for testing. Casings of the same diameter as in Example 4 were then made from this web in the manner set forth in Example 1 and were tested. The test data is given in Table II.

#### EXAMPLE 6.

The procedure of Example 5 was repeated using 1.5% by weight of "Kymene 557". In each example the diameters of the casings tested were the same. The results are set forth in Table II.

From Table II it is apparent that improved

- 5 burst strengths were obtained for casings made in accordance with the present invention, the webs used to make these casing again having appreciably lower tensile or breaking strengths than the webs made according to the inventional method. This would appear to indicate that improved bonding between the paper and the cellulose coating is accomplished according to the present invention, particularly when it is noted that in the Underwood patent burst strength increased only with an increase in tensile strength. 10

TABLE II

Test	Example 4	Example 5	Example 6
<u>Wet Breaking Strength</u>			
(g/in width)			
Machine Direction —	1414	656	650
Cross Direction —	1119	636	662
<u>Dry Breaking Strength</u>			
(g/in width)			
Machine Direction —	6500	2247	2297
Cross Direction —	4150	2040	2110
<u>Density (g/cc)</u>	0.351	0.184	0.184
<u>Burst Pressure (mm)</u>	428	485	478
<u>Ream Weight (lbs)</u>	13.7	13.9	13.8
<u>Burst Ratio</u>	31.2	35.0	34.6
<u>Improvement in Burst Strength</u>	—	12.18%	10.90%

- 15 EXAMPLES 7 AND 8  
The procedure of Example 3 was followed using 1.0% of "Kymene" in Example 7 and

1.5% of "Kymene" in Example 8. The tests results are set forth in Table III.

TABLE III

Test	Example 7	Example 8
<u>Wet Breaking Strength</u> (g/in width)		
Machine Direction	658	869
Cross Direction —	683	714
<u>Dry Breaking Strength</u> (g/in width)		
Machine Direction —	2212	3131
Cross Direction —	1950	2447
<u>Density (g/cc)</u>	0.230	0.265
<u>Burst Pressure (mm)</u>	473	452
<u>Ream Weight (lbs)</u>	12.6	12.2
<u>Burst Ratio</u>	37.2	36.9

EXAMPLE 9  
(Comparative)

5 A base web and casing was made using the procedure of Example 1 except that a different source of hemp fibers was employed. Samples were taken in the usual manner and

tested, the test data being given in Table IV.

EXAMPLE 10

The procedure of Example 3 was repeated using as a furnish hemp fibers from the same source as Example 9. The data obtained is set forth in Table IV.

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TABLE IV

Test	Example 9	Example 10
<u>Wet Breaking Strength</u>		
(g/in width)		
Machine Direction —	1304	617
Cross Direction —	1221	563
<u>Dry Breaking Strength</u>		
(g/in width)		
Machine Direction —	4883	2217
Cross Direction —	3883	1758
<u>Transverse Elongation</u>		
Edge (% elongation)	2.39	3.11
Center (% elongation)	0.77	1.89
Reduction	—	24.7%
<u>Density (g/cc)</u>	0.265	0.267
<u>Burst Pressure (mm)</u>	b	435
<u>Ream Weight (lbs)</u>	12.45	12.53
<u>Burst Ratio</u>	—	34.7

a — Elongation results were obtained using a 2.5 pound load.

b — Data not available.

#### WHAT WE CLAIM IS:—

1. A method of producing reinforced films suitable for packing meat products comprising the steps of forming a dilute dispersion of cellulosic fibers and depositing said dispersed fibers in the form of a continuous web while incorporating an alkaline-curing synthetic resin, impregnating the web with a caustic cellulose solution; the web having a density prior to impregnation of at least 0.15 g/cc as determined by TAPPI test method T411—m—44; regenerating the cellulose impregnating said web; and thereafter drying the impregnated web to provide a reinforced film-like material.
2. A method as claimed in claim 1, wherein the curing resin content is up to 10% by weight based on the solids within the furnish.
3. A method as claimed in claim 1 or 2, wherein the resin content is up to 5.0% by weight based on the solids within the furnish.
4. A method as claimed in any one of

the claims 1 to 3, wherein the curing resin is a polyethylene imine or a reaction product of epichlorohydrin and a polyamide containing secondary amine groups.

5. A method as claimed in any one of the claims 1 to 4, wherein the web has a density prior to impregnation of less than 0.35% g/cc as determined by TAPPI test method T411—m—44.

6. A reinforced film suitable for packing meat products comprising a fibrous base web impregnated with regenerated cellulose, said base web comprising essentially cellulosic, randomly interconnected fibers and having an alkaline-curing synthetic resin incorporated therein and a density prior to impregnation of at least 0.15 g/cc, as determined by TAPPI test method T411—m—44.

7. A reinforced film as claimed in claim 6, wherein the curing resin content is up to 10.0% by weight based on the solids in the web.

8. A reinforced film as claimed in claim



- 6 or 7, wherein the resin content is up to 5.0% by weight based on the solids in the web.
- 5 9. A reinforced film as claimed in any one of the claims 6 to 8, wherein the resin is a polyethylene imine or a reaction product of epichlorohydrin and a polyamide containing secondary amine groups.
- 10 10. A reinforced film as claimed in any one of the claims 6 to 9, wherein the web possesses a density of less than 0.35 g/cc as determined by TAPPI test method T411—m—44, the said density being the density for the web prior to impregnation.
- 15 11. A reinforced film as claimed in claim 9, wherein the resin content is 0.5% to 3.0% by weight based on the solids in the web.
12. A method of producing a reinforced film as claimed in any one of the claims 1 to 5, substantially as herein described. 20
13. A reinforced film when produced by the method as claimed in claims 1 to 5 and 12.
14. A reinforced film as claimed in any one of the claims 6 to 11, substantially as 25 herein described.

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Chartered Patent Agents,  
Agents for the Applicants.

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